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## The Effect of Carbon Type and Processing on High Rate Thionyl Chloride Cathode Collectors

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### Abstract

This study was initiated to determine the effects of carbon type, treatment and additives and the manufacturing process on high rate performance of thionyl chloride cathode collectors. Seventeen different collector materials and treatments were tested in demountable test cells and the results compared on the basis of capacity and energy per unit void volume of the cathode collector. Superior results were consistently obtained from CoTMPP catalyzed carbons and from carbon microfibers. Better voltage results were obtained from Ketjenblack EC, but the capacity was not consistently better than acetylene black. Process conditions did not show strong correlations, but in some cases there appears to be a best process for a given carbon type. A significant high rate (7 A test) capacity improvement was seen in F cells with a carbon fiber electrode in agreement with test cell results. Other test cell results did not correlate well with F cell tests, but further work is required to establish statistical significance.

### Introduction

The effect of the carbon type and the cathode collector manufacturing process on the high rate performance of thionyl chloride cathode collectors has been the subject of several investigations. In early work, Dey (1,2) found little effect of using carbon blacks with a high BET surface area. Work by Christopulos and Gilman (3) showed the same lack of correlation with BET surface area but found the Coulombic capacity proportional to the initial volume available for solid discharge products, the collector pore volume. Although the discharge capacity has been found to be correlated to the DBT absorption number for most carbons (4), the capacity may be controlled by the porosity since the porosity was found to be proportional to the surface area. However, other investigations of liquid cathode cell performance found that the highest surface area carbons gave the highest discharge capacity (5-8). The use of additives, catalysts and pretreatments has also been reported to improve high rate capacity. Walker et al. (8) used acetone as an extractant and found

In a preliminary report, Bailey and Blomgren (11) compared several methods of high rate capacity improvement. Those results have been replicated to enable statistical comparisons and the study has been expanded to include a number of new carbon and catalysts types. Also, in order to determine if the advantages found in the test cells could be obtained in sealed automatically manufactured cells, the most promising of these carbon materials were evaluated in high area F size cells.

## Materials

Battery grade acetylene black (Chevron Chemical Co.) was used as the standard material, while polytetrafluoroethylene (PTFE) as T30B colloidal and 6C fine particle TEFLON (du Pont) were used as binder materials. A steam activated carbon of high surface area and heat treated CoTMPP on acetylene black were obtained from Eltech Systems Co. Acetylene black was soaked overnight in reagent grade acetone or toluene for the Wash 1 or Wash 2 samples, filtered and dried at ambient. Ketjenblack EC (Akzo Chemie), Vulcan XC-72 (Cabot Corp.), and Black Pearls 2000 (Cabot Corp.) were used as purchased. The carbon fiber material was a special carbon microfiber. Some of the carbons were catalyzed with CoTMPP by Dr. S. L. Gupta at Case Western Reserve University for our studies using their standard precipitation and heat treatment process (12).

In this study the standard method to produce collectors from these materials (Process 1) was to blend the carbon powder with 6C PTFE, and to knead the blended solid with mineral spirits. The kneaded carbon-PTFE dough was then calendered to form a film. After drying, the film was re-rolled to produce a collector approximately 0.25 mm thick with a porosity of approximately 80%. The collector sections produced by this method were flexible and easily handled. Process 1A is a variation in which the carbon-PTFE dough is dried, granulated, then rewet with mineral spirits and calendered into a film. Process 2 used a conventional blend of T30B solution and the carbon. The mix was calendered and rolled wet prior to drying. Process 3 used dried and granulated material from Process 2, blended it with mineral spirits and rolled the

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material while moist. Process 4 simply used the collector material from F cells (13), scraping the carbon from the metal mesh support. Process 5 used an alcohol wash, which collapses the carbon, followed by overnight air drying and then Process 1.

These collector sections were evaluated in the test cell shown in Fig. 1. This test cell was built to mimic many of the features of a sealed or finished high area cell. These features include excess anode and electrolyte (such as would be found in a finished cell discharged at a high rate) so that the capacity is limited by the cathode collector. The electrodes are placed under a modest compression at a fixed height rather than spring loaded. As a result, the pressure between the electrodes decreases during discharge. A second important factor in the cell design is that the electrodes communicate with the excess electrolyte of the cell only at the ends of the electrodes. As the discharge proceeds, the pressure between the electrodes diminishes and the excess electrolyte can flow into the cell from the exposed ends of the electrodes and separators. However, the chemical composition of the electrolyte is expected to change in a way similar to a finished cell because of the restricted accessibility of the excess liquid to the interelectrode region of the cell. These features of the test cell are believed to correspond to actual conditions in most battery designs and differ from most cells which have been employed previously to test cathodes for thionyl chloride cells.

With reference to Fig. 1, the main cell electrode stack, which consists of a piece of nickel foil, the free standing carbon-PTFE film, two layers of separator paper, and a lithium anode, is placed in the groove of the TEFZEL cell body. This electrode stack is then held in place by a TEFZEL top and compressed by a projection which fits into the groove in the cell body. A lithium reference electrode is inserted between the two separator papers at the end of the cell. In the assembled view of the cell, one can see the relative position of the reference electrode in between the anode and cathode. Once the cell is assembled, it is placed in a jar and flooded with 1.5M  $\text{LiAlCl}_4$  in thionyl chloride electrolyte. In order to measure the cathode polarization, the cell was put under a resistive load and the anode-cathode and cathode-reference potentials read after 10 seconds. The cell capacity (limited by the cathode collector) was measured at 20 mA/cm<sup>2</sup> and 10 mA/cm<sup>2</sup> by discharging the cell through resistive loads to a cutoff voltage of 2 V. Three to five cells were tested under each condition to permit statistical comparisons.

#### Evaluation Using F cells

An "F" sized jellyroll cell previously described by Ayers et al (13) was used in all testing. This cell is shown in Fig. 2. This cell was selected because it is supported by a large data base and is capable of delivering high currents. The step between the test cell and sealed, finished high rate F cells is important for verifying results.

Some procedures such as cathode collector manufacture and jellyroll winding are critical to service reproducibility. Pilot line equipment was used where possible to improve cell consistency. The cells made for this study had two modifications as compared to the standard "F" cells. The first was a modified mixing process. Unfortunately, all the carbons were not available in large enough amounts to use the pilot line mix equipment. The modification was to change from a damp mixed PK Blender process to a slurry mixed Hobart mixing (Kitchen-Aid, Model #K5-A). The rest of the process stayed the same: the wet mix is dried overnight at 110°C, then densified and granulated. Thus we were able to test materials with as little as 150 grams and still have enough mix to use the pilot line cathode collector molding equipment. On the pilot line equipment, the mix was pressed onto a perforated electrode carrier, and once sintered, was ready for winding.

The second modification was changing the anode from two 0.006 in. thick pieces of lithium to one 0.005 in. and one 0.010 in. thick piece of lithium pressed onto a metal carrier. The purpose was two fold. First, the cathode collector is the final wrap which leaves an extra half thickness of cathode collector for the anode to discharge against. The anode opposite this extra cathode is typically totally used at the 2 A rate. To insure that the cell was not anode limited in this area, the outer portion of the anode was substantially thickened. Secondly, if more anode was available in this portion, the service was expected to increase.

Cells were discharged at a constant current rate of 2 and 7 A as fresh cells. Cell temperature was recorded for the 7 A cells by a thermocouple taped to the middle of the cell wall.

## Results and Discussion

### Test Cell Results

In order to compare the cathode collectors on an equal basis and remove any differences caused by differences in cathode thickness or porosity, the capacity and energy of the electrodes is compared based on the void volume of the electrode. This procedure is based on the theory that the maximum capacity of a cathode collector is limited by the volume of the voids which can contain the insoluble discharge products, LiCl and S. The volume of these materials should limit the capacity of a collector to 1100 mAh/cc of void.

Seventeen carbons were compared using the standard process. The first 5 types were based on Chevron acetylene black. They were: Chevron acetylene black, steam activated Chevron acetylene black, Chevron acetylene black extracted with acetone, Chevron acetylene black which had been extracted with toluene and Chevron acetylene black to which 20 % carbonyl Ni powder had been added. The remaining materials investigated were Ketjenblack EC, Ketjenblack EC plus 20% carbonyl Ni powder, Black Pearls 2000, Cabot XC-72, and a fiber like carbon. Two of these carbons, Chevron acetylene black and the fiber like carbon were heat treated

at 3000 °C for 1 hour to modify the carbon structure. Four of the carbons were heat treated with CoTMPP to catalyze the material. These were Chevron acetylene black, Ketjenblack EC, Black Pearls 2000 and the fiber like carbon.

The energy in mWh/cc void and capacity in mA·h/cc void for the first seventeen collector types are summarized in Figures 3 and 4. Standard deviations are shown at the top of each bar. These figures show that Ketjenblack-Ni, CoTMPP catalyzed and carbon fiber electrodes have improved performance when compared to the acetylene black control. Two of the high surface area carbons, XC-72 and Black Pearls 2000, and the solvent washed carbons show poorer performance. Table 1 gives the percent change from acetylene black for the materials which show significant differences. The improvements found for the catalyzed materials are believed to be due to changes in the carbon surface. They generally result in an increased operating voltage as well as an increased capacity. The Ketjenblack-Ni electrode shows an increased voltage but little change in capacity. To clarify these factors, the quasi steady state polarization curves and discharge curves comparing several of the Chevron acetylene black based carbons with the Ketjenblack, carbon fiber and cobalt catalyzed acetylene black for representative cells are shown in Figures 5 and 6. In the case of the carbon fiber containing electrodes, the substantial improvement is believed to be due to the physical nature of the electrode, rather than the energetics of the active sites, since the polarization and operating voltage are more like acetylene black than the catalyzed materials. The high aspect ratio of fibers should enhance the electrical conductivity of the electrode and change the nature of the pore shapes and sizes. An improvement is still present when used as a 50-50 mixture with the acetylene black. Heat treated fibers and cobalt catalyzed fibers did not perform as well as the untreated fibers. The significant decrease found for the two high area carbons, Ketjenblack EC and Black Pearls 2000, is believed to result primarily from the way in which they handle with the process used in these tests. These materials have a tendency to form an electrode with a smooth closed surface. This electrode structure is believed to block off with discharge products at the surface when discharged at high rates.

The effect on the energy and capacity per cc of void for several different processes is summarized in Figure 7 and Table 2. The significance of the results is not clear, but it was found that Process 2 was inferior to Process 1 for acetylene black, but superior for acetylene black with 20% nickel, while Process 3 was the same as Process 1 for acetylene black. Process 3 gave somewhat better results for Ketjenblack at the higher rate, but the lower rate results were not significantly different, although both rates performed better than Ketjenblack with Process 1. Improvement was found for the fiber like carbon with Process 1A compared to acetylene black, but these improvements were seen with Process 1 as well and are probably more a result of the carbon type. Thus, we conclude that for most carbon materials the capacity and energy of the collector is not sensitive to the collector manufacturing

process. This appears not to be the case for very high surface area carbons, however, which must be carefully prepared so as not to densify the surface too much.

The effect of catalyst loading and electrode thickness are shown in Figure 8 and Table 3. The catalyst loading at three different levels did not show significant differences in the energy or capacity, but, as seen in Figure 9, the discharge occurs in two plateaus for the lower loadings. Apparently, the high energy sites of the cobalt are filled at the lower loading before the end of discharge and the rest of the discharge occurs on the carbon, at the carbon voltage. The effect of increasing thickness of the electrode was to lower the capacity and energy for unit void volume (the capacity per unit area actually increases), but by modifying the process to Process 5, a 0.050 in. electrode performed like the 0.010 in. thick control. In this case, Process 5 is believed to have provided a more favorable pore structure.

#### F Cell Results

"F" cells were made with a number of cathode materials from the test cells. A total of eight different cathode collectors were evaluated: 70:20 (wt%) acetylene black :nickel powder; 90 (wt%) AB; 90 (wt%) toluene washed AB; 90 (wt%) heat treated AB; 90 (wt%) CoTMPP catalyzed AB; 90 (wt%) Ketjenblack EC; 70:20 (wt%) Ketjenblack EC:nickel powder; and 90 (wt%) carbon fiber. Each cathode collector had 10 (wt%) Teflon (T30B emulsion) as the binder. One of the carbons, the CoTMPP catalyzed carbon, could not be processed so that it adhered to the cathode collector carrier, and hence could not be tested.

Scrap and overusage is a significant factor in using pilot line equipment for small experimental lots. Frequently a mix lot of 10-12 cells was decreased to 1-3 cells through cell testing. Where a limited number of cells was available at testing, the 7 A rate was given the priority. Additionally, as the cathode collectors were potentially energetic, care was taken to insure safety in manufacturing and testing of the cells.

A summary of the cells tested at 2 and 7 A is presented in Table 4 and Figure 10 with statistical comparison to the control where possible. The outstanding cathode collector was the fiber carbon which outperformed the control at 7 A by 34%. This improvement was statistically significant to the 99% confidence level. Only the carbon fiber shows a better capacity at the 7 A rate than at the 2 A rate. The discharge curves of the control and the carbon fiber show an unusual effect giving rise to the improvements (Figures 11 and 12). The carbon fiber shows a distinct second hump which is coincident with the increase in temperature near the end of cell life. This hump is not seen in the control. The problem with this extra service is the dip in voltage which gets very close to the 2 volt cutoff. Two other comments are important in the curve comparison. Note that the fiber-like carbon shows a 0.2 volt lower CCV and also runs at a

20°C lower operating temperature during discharge.

#### Comparison of F Cell and Test Cell Results

The test cell and F cell data can be compared on the basis of the capacity per cc of void volume. The values calculated for the total F cell cathode collector at the 7 A rate (about 13 mA/cm<sup>2</sup> on average) and for the test cell at 10 mA/cm<sup>2</sup> are shown in Figure 13. Somewhat surprisingly, the carbon fiber results in the F cell tests are not as good as the acetylene black when compared on this basis, but the higher porosity of the electrode results in the absolute advantage of the fibers discussed above. Also, the results for the F cell and test cell do not correlate well. The F cell output is relatively constant while the test cell data appears to depend more heavily on the carbon used. From this we tentatively conclude that an important limitation on the F cell is in the cell or electrode construction while the collector carbon has a secondary or interactive effect, although more replications of the F cells should be made for statistically sound comparisons. The two types of cells can also be compared on the basis of capacity per gram of mix under the similar test conditions, as shown in Figure 14. Again no clear correlation exists between the two types of cells, but in this comparison the carbon fiber collector is superior to the acetylene black controls (both with and without nickel) for both F cells and test cells.

#### Conclusions

Seventeen different collector materials and treatments were tested in demountable test cells and the results compared on the basis of capacity and energy per unit void volume of the cathode collector. Superior results were consistently obtained from CoTMPP catalyzed carbons and from carbon microfibers. Better voltage results were obtained from Ketjenblack EC, but the capacity was not consistently better than acetylene black. Process conditions did not show strong correlations, but in some cases there appears to be a best process for a given carbon type. A significant high rate (7 A test) capacity improvement was seen in F cells with a carbon fiber electrode in agreement with test cell results. In general, however, capacity per unit void volume from test cells did not correlate well with F cell service although the statistical basis was poor because of small F cell sample size. We were unsuccessful in making F cell electrodes using the CoTMPP catalyzed materials with our methods, so we were unable to make this comparison. Although the catalyst material is expensive, it may be worth efforts to find ways to process these materials into strip electrodes for larger cells for some applications.

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## Figure Titles

Figure 1. Exploded View of demountable test cell.

Figure 2. Cutaway view of F cell with enlargements of glass to metal seal and fill tube regions.

Figure 3. Capacity per unit void volume (a) and energy per unit void volume (b) for various carbons at two current densities as measured in test cells. Standard deviations are shown at the top of the bars. AB is Chevron acetylene black, ACTIV is a steam activated acetylene black, CO-CAT is acetylene black with 10% CoTMPP catalyst, GRAPH is a high temperature heat treated acetylene black, WASH1 and WASH2 are acetylene black extracted by acetone and toluene, respectively, AB-NI is a 70:20 acetylene black:nickel powder mix, KET is Ketjenblack EC and BP is Black Pearls 2000.

Figure 4. Capacity (a) and energy (b) per unit void volume in test cells for various carbons. XC72 is Vulcan XC-72, CO-XC72 is a 10% CoTMPP catalyzed Vulcan XC-72, KET-NI is a 70:20 Ketjenblack EC:nickel powder mix, FIBER is a carbon fiber and the HT is high temperature heat treated, AB is 1:1 with acetylene black, and CO is 10% CoTMPP catalyzed. CO-BP is 10% CoTMPP catalyzed Black Pearls 2000.

Figure 5. Quasi steady state polarization as a logarithmic function of current density measured in test cells for acetylene black, toluene washed acetylene black, carbon fibers, Ketjenblack EC, and 10% CoTMPP catalyzed acetylene black.

Figure 6. Discharge curves for same samples as Figure 5 at 20 mA/cm<sup>2</sup> showing full cell potential of test cells.

Figure 7. Capacity (a) and energy (b) per unit void volume in test cells for various carbons undergoing various processes (1-5) described in text. Carbon designations as in Figure 3 and 4.

Figure 8. Capacity (a) and energy (b) per unit void volume in test cells comparing electrode thickness and CoTMPP loading. Second designation after AB is the electrode thickness in mils. Second designation after CO is the % initial loading of the catalyst. CO-2/10% is a Vulcan XC-72 carbon instead of AB. CO-2/2.6 is AB with 2.6% loading made by process 2.

Figure 9. Discharge curve in a test cell of 2.6% CoTMPP on acetylene black made with process 1 at 10 mA/cm<sup>2</sup>. ..... is the anode to cathode voltage, ----- is the lithium reference to cathode voltage.

Figure 10. Capacity of various collector materials tested in

F cells at 2 and 7 A constant current.

Figure 11. F cell discharge curve and external cell temperature at 7 A constant current for 70:20 acetylene black:nickel powder cathode collector.

Figure 12. F cell discharge curve and external cell temperature at 7 A constant current for carbon fiber cathode collector.

Figure 13. Capacity in mAh/cc void for F cell at 7 A and test cells at 10 mA/cm<sup>2</sup> for various carbons. Collector designations as in Figures 3 and 4.

Figure 14. Capacity in Ah/g of mix for F cells at 7 A and test cells at 10 mA/cm<sup>2</sup> for various carbons as in Figure 13.

Table 1

Comparison of Significant Differences of Test Carbons from  
Acetylene Black (AB)

<u>Collector Material</u>	<u>Current Density (mA/cm<sup>2</sup>)</u>	<u>% Differences</u>	
		<u>Capacity</u>	<u>Energy</u>
CoTMPP on AB	10	+21	+36
	20	+45	+67
Acetone washed AB	10	-20	-17
	20	---	---
Toluene washed AB	10	-14	-11
	20	-15	---
Black Pearls 2000	10	-36	---
	20	-72	-74
Cabot XC-72	10	-27	---
	20	-46	-45
CoTMPP on XC-72	10	+33	+48
Ketjen EC + 20% Ni	10	+8	+20
	20	---	+29
Carbon fiber	10	+67	+76
	20	+69	+79
Heat treated carbon fiber	10	+26	+27
	20	+24	+29
1:1 AB:carbon fiber	10	+20	+36
	20	---	---
CoTMPP on carbon fiber	10	+41	+29
	20	+58	+62
CoTMPP on Black Pearls 2000	10	-15	---
	20	+11	+24

Note: significant differences based on student t-Test at 95% Confidence level.

Table 2  
Comparison of Significant Differences from  
Acetylene Black (AB) Using Process 1

<u>Material/Process</u>	<u>Current Density (mA/cm<sup>2</sup>)</u>	<u>% Differences</u>	
		<u>Capacity</u>	<u>Energy</u>
AB/2	10	-33	-36
	20	-28	-24
80% AB-20% Ni/2	10	+32	+38
	20	+44	+52
Ketjen/3	20	+26	+40
Fiber/1A	10	+48	+93

Note: significant differences based on student t-Test at 95% confidence level. Processes described in text.

Table 3

Electrode Thickness Comparison for Acetylene Black

<u>Material</u>	<u>Current Density (mA/cm<sup>2</sup>)</u>	<u>% Differences</u>	
		<u>Capacity</u>	<u>Energy</u>
Compared with AB-0.010 in.			
AB-0.035 in.	10	-40	-37
	20	-48	-44
AB-0.050 in.	20	-49	-44
AB-0.050 in./Process 5	10	Same	Same
	20	Same	Same

Note: significant differences based on student t-Test at 95% confidence level. All tests except last used Process 1.

Table 4

## Comparison of Cathode Collectors for High Rate Service in P Cells

<u>Cell Description</u>	<u>2A Test</u>				<u>7A Test</u>			
	<u>Average Service (Ah)</u>	<u>Number of Cells</u>	<u>Standard Deviation</u>	<u>Student t-Test Significance</u>	<u>Average Service (Ah)</u>	<u>Number of Cells</u>	<u>Standard Deviation</u>	<u>Student t-Test Significance</u>
Control	12.38	7	0.21		11.51	5	0.65	
70:20 AB:W								
AB	12.80	1	-		12.30	1	-	
Toluene washed AB	13.20	1	-		12.18	2	0.10	Not different 95% confidence
Heat treated AB	12.40	1	-		11.20	1	-	
CoTMP catalyzed AB	Not tested				Not tested			
Ketjen EC	Not tested				10.43	2	0.10	Not different 95% confidence
70:20 Ketjen:W	Not tested				8.05	1	-	
Fiber	12.50	1	-		15.43	2	0.02	Different with 99% confidence
1:1 Fiber:AB	12.53	2	0.23	Not different 95% confidence	11.30	2	0.05	Not different 95% confidence



Figure 1

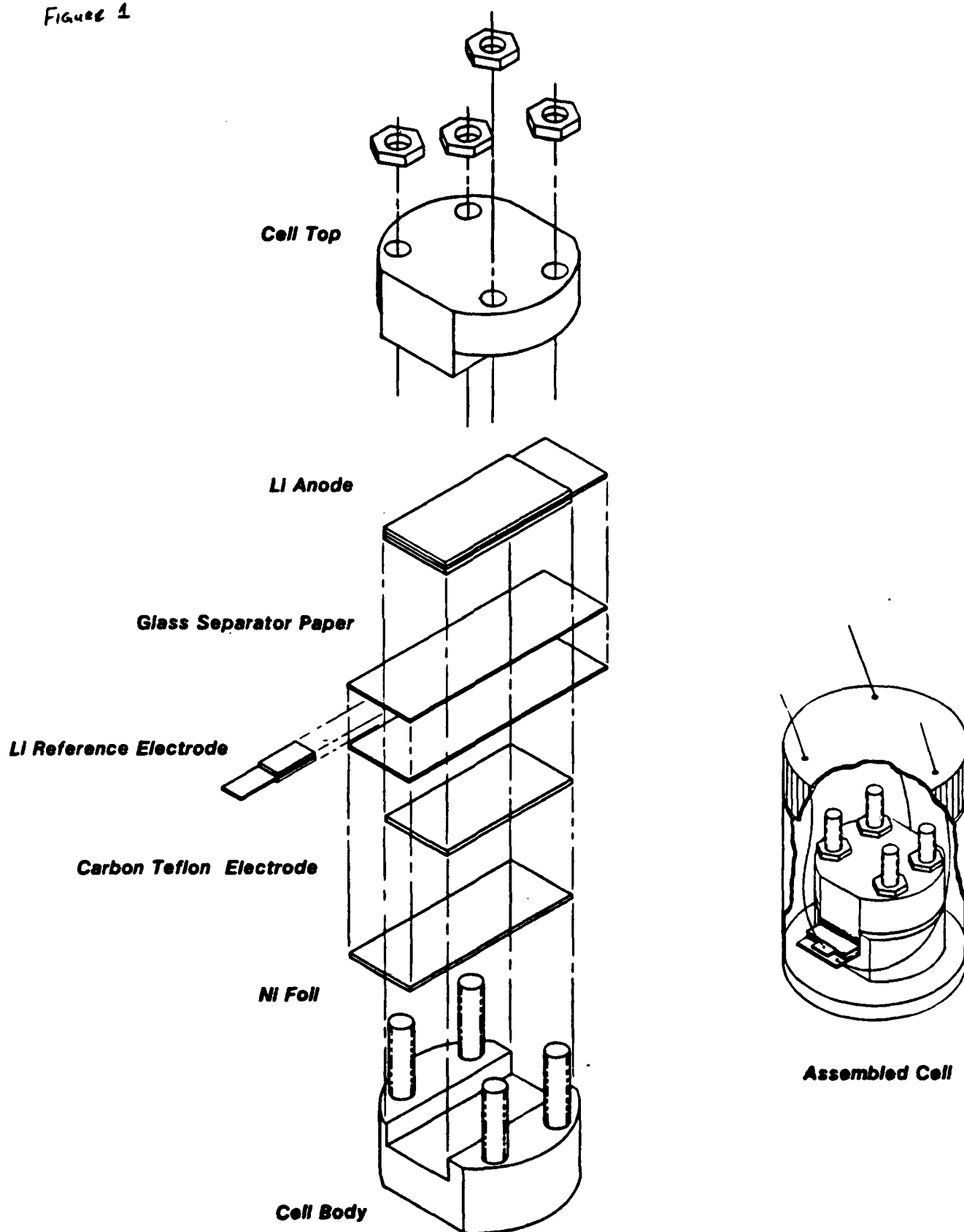
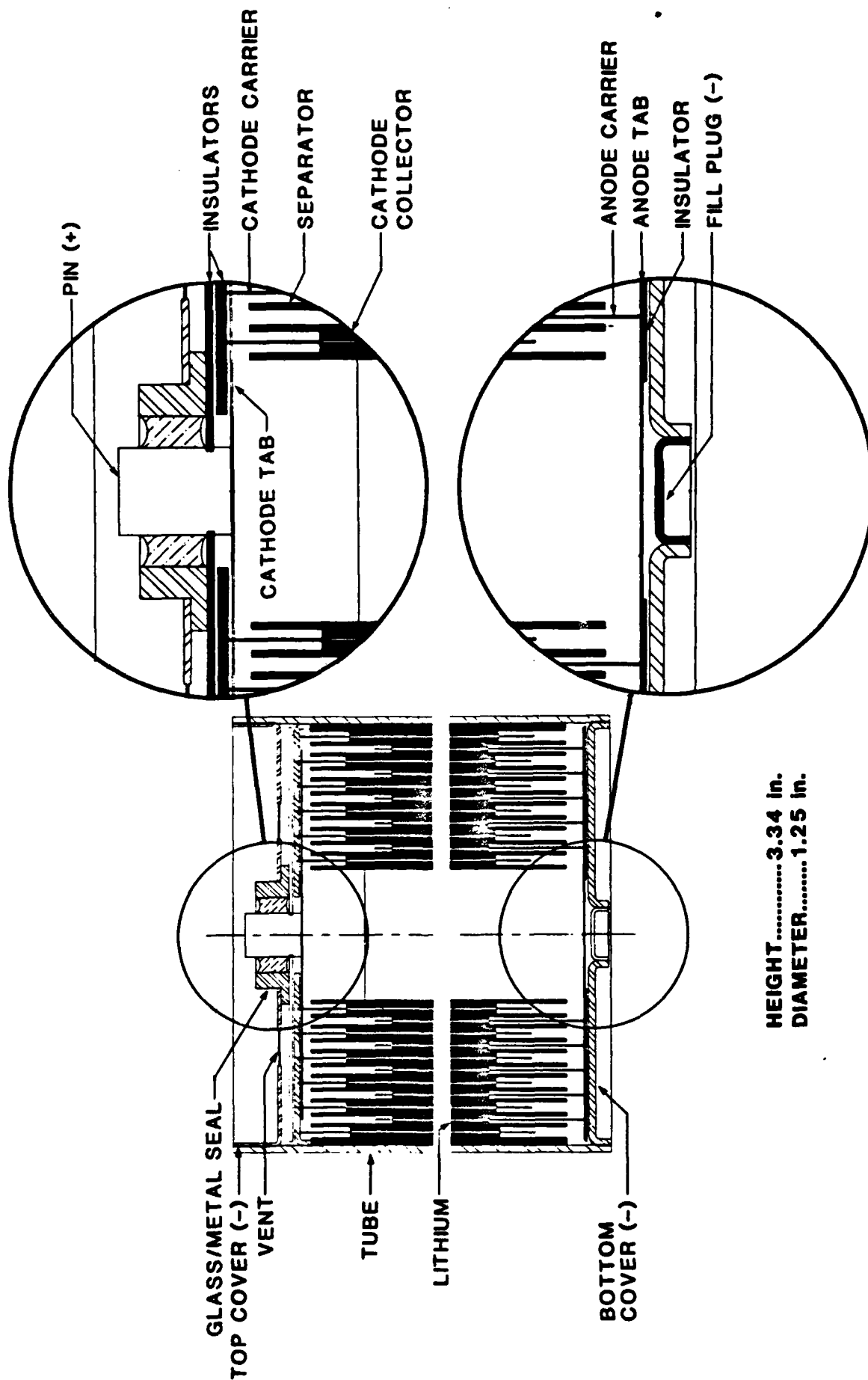


Fig 2



HEIGHT ..... 3.34 in.  
DIAMETER ..... 1.25 in.

Fig. 3a

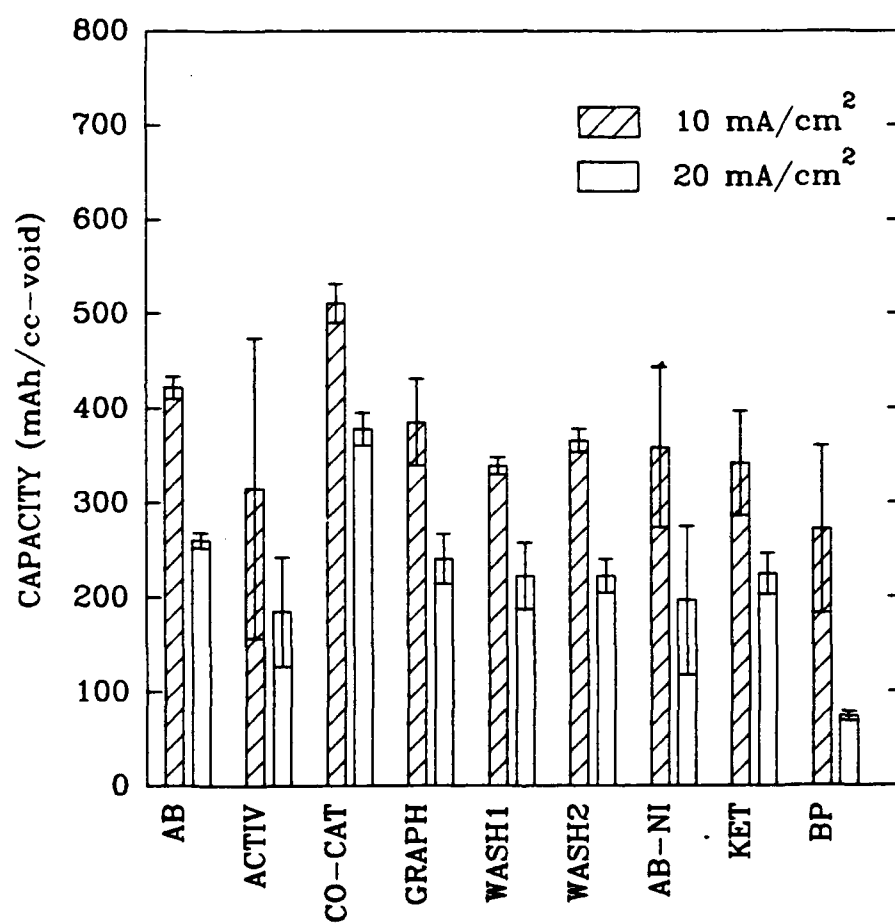


Fig. 3b

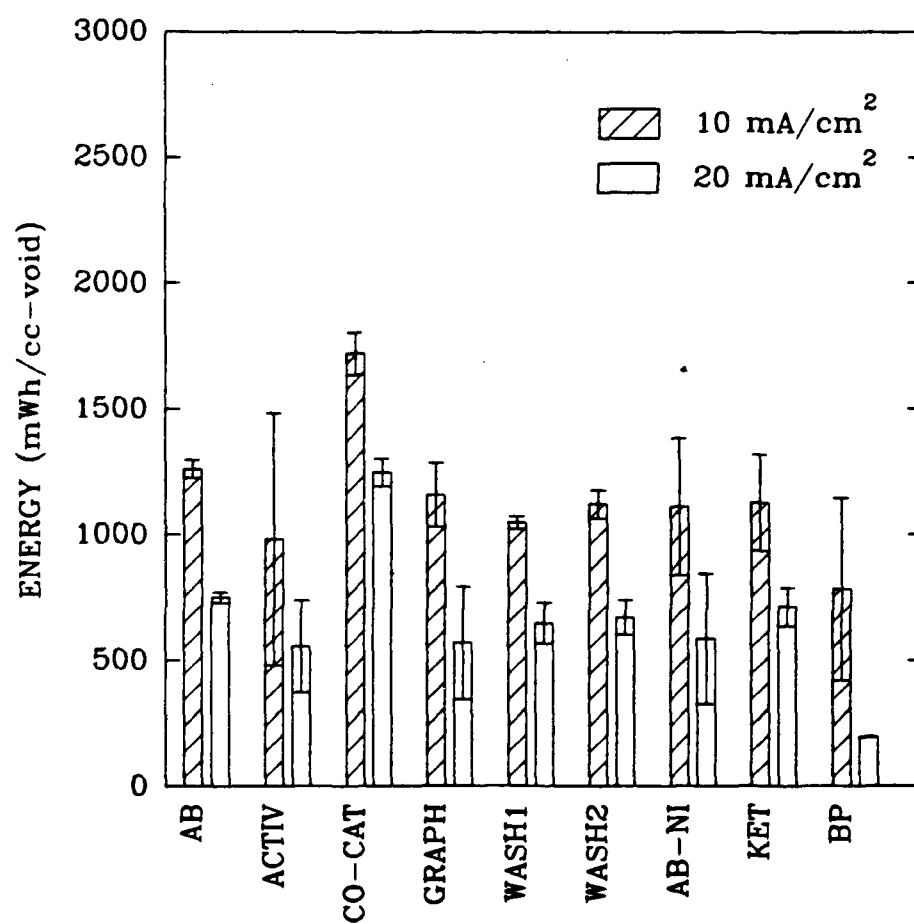


Fig. 4a

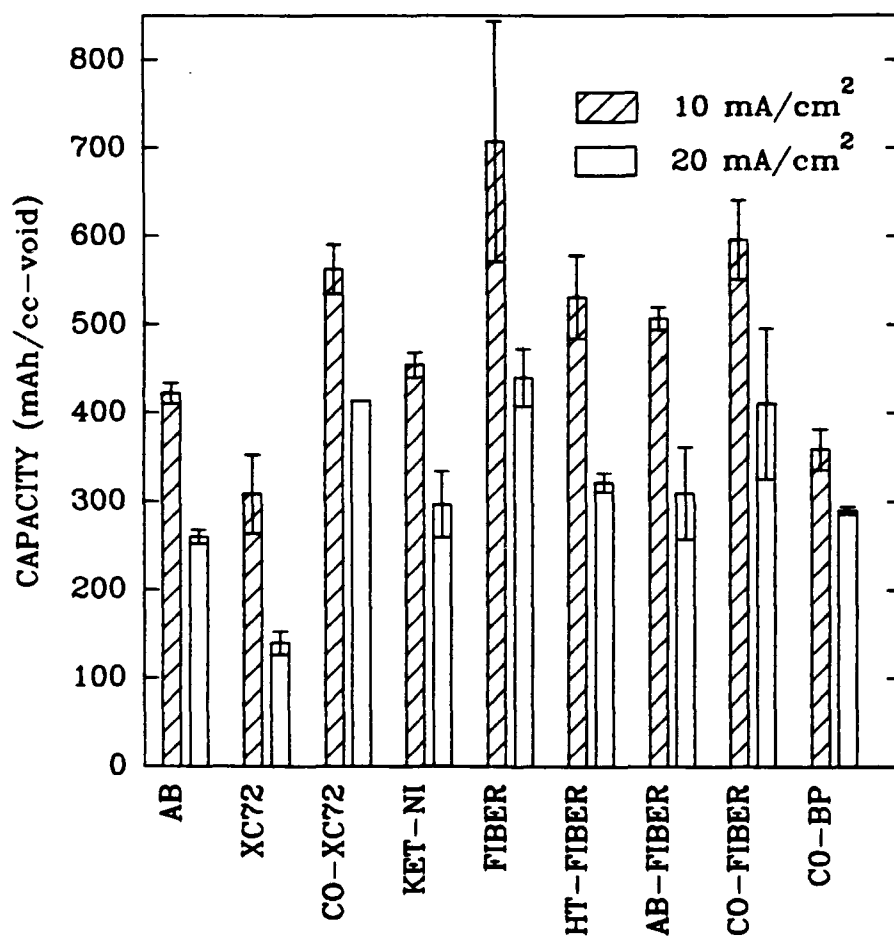


Fig. 4b

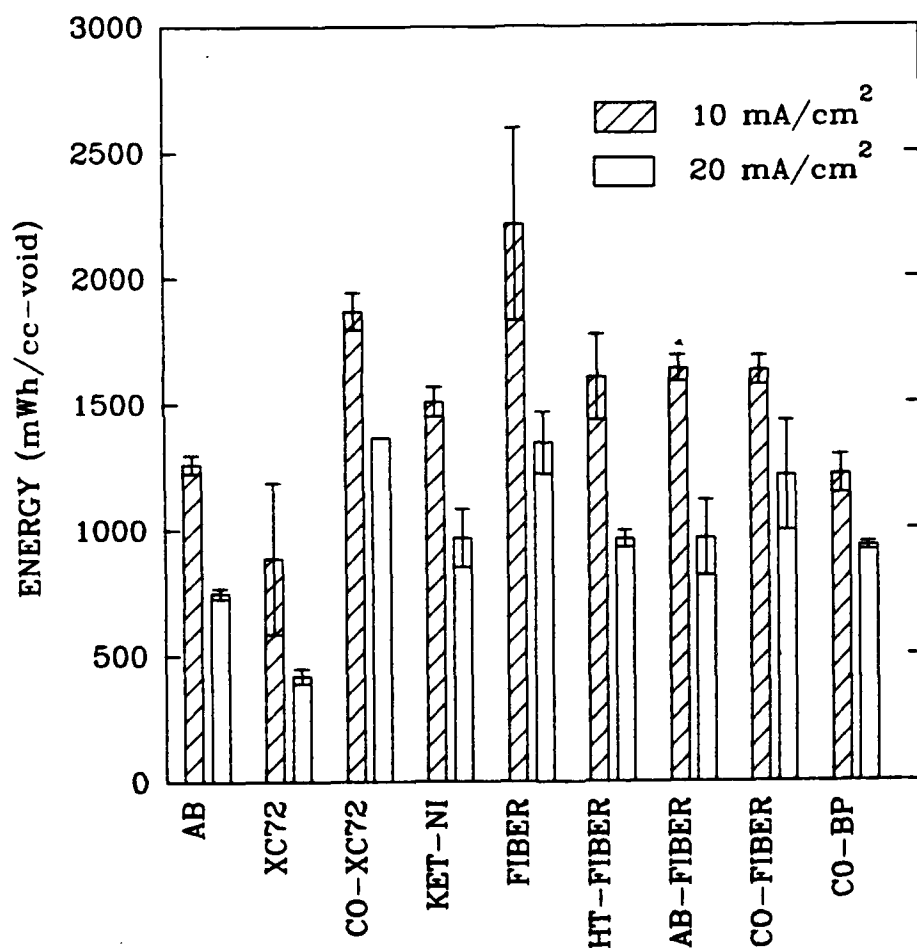


Fig. 5

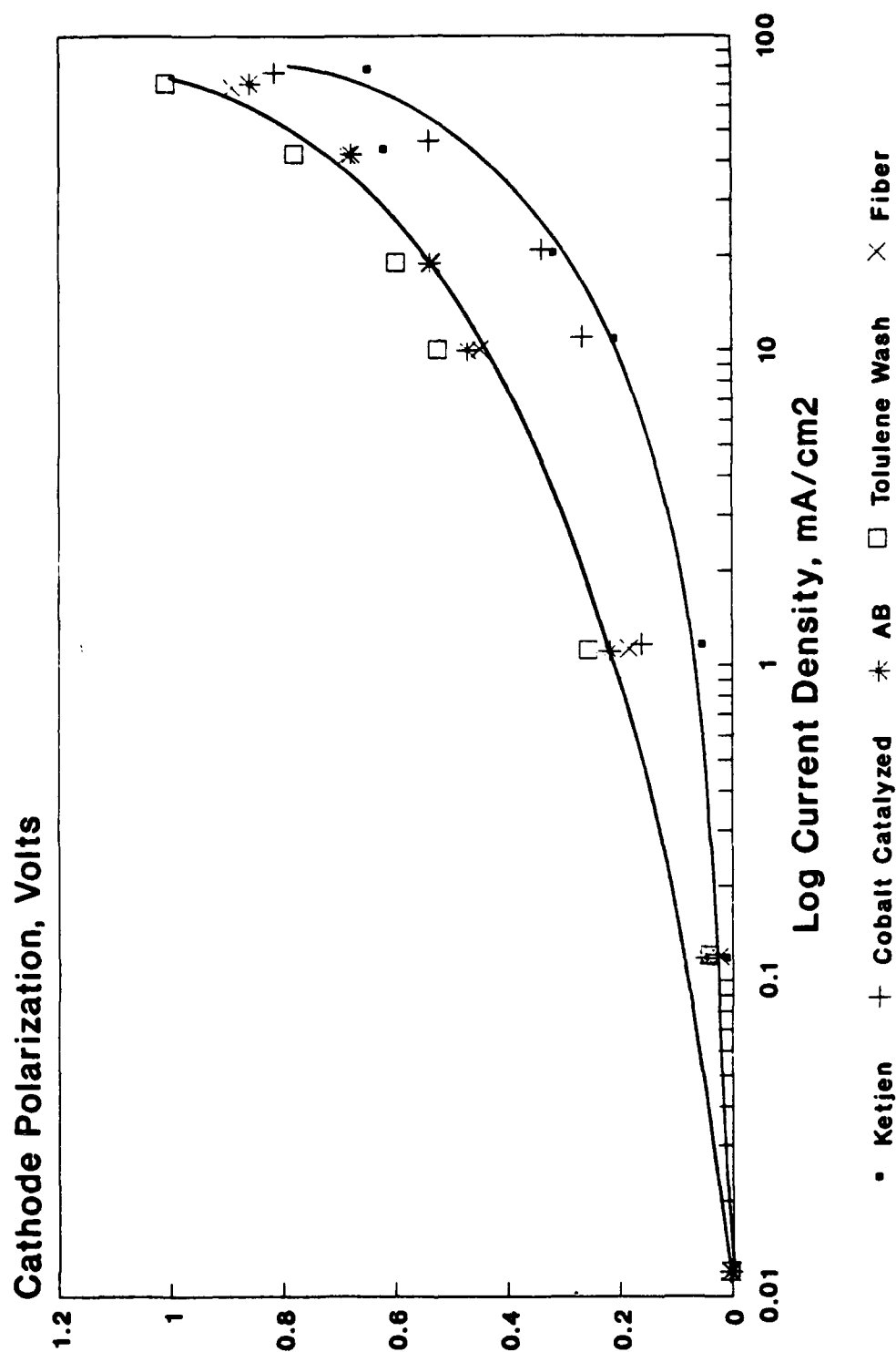


Fig. 6

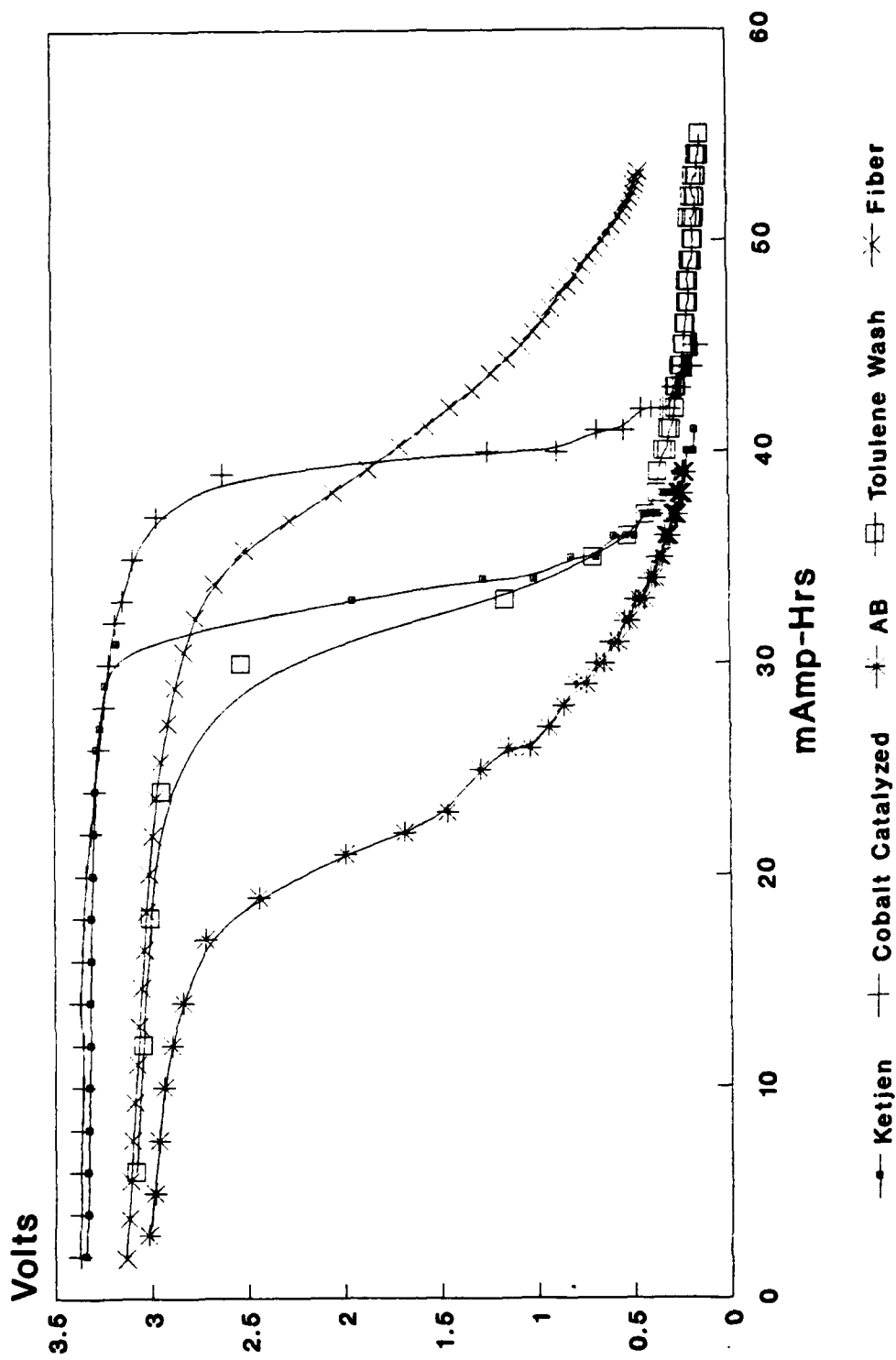




Fig. 7a

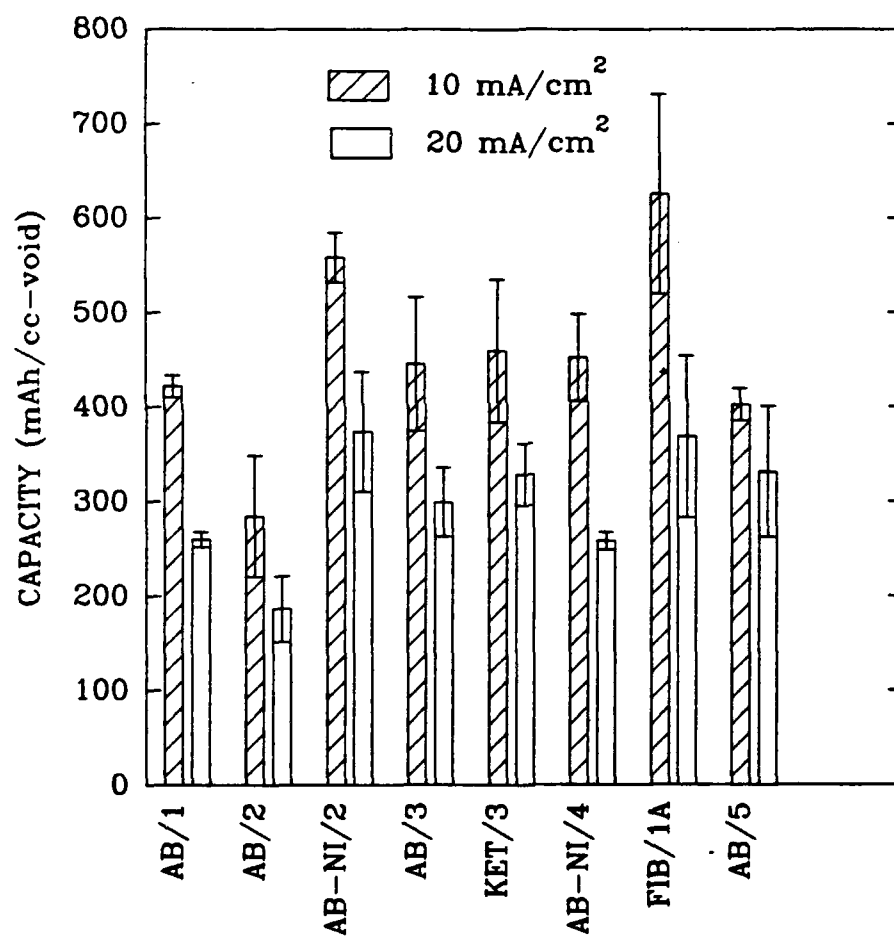


Fig. 7b

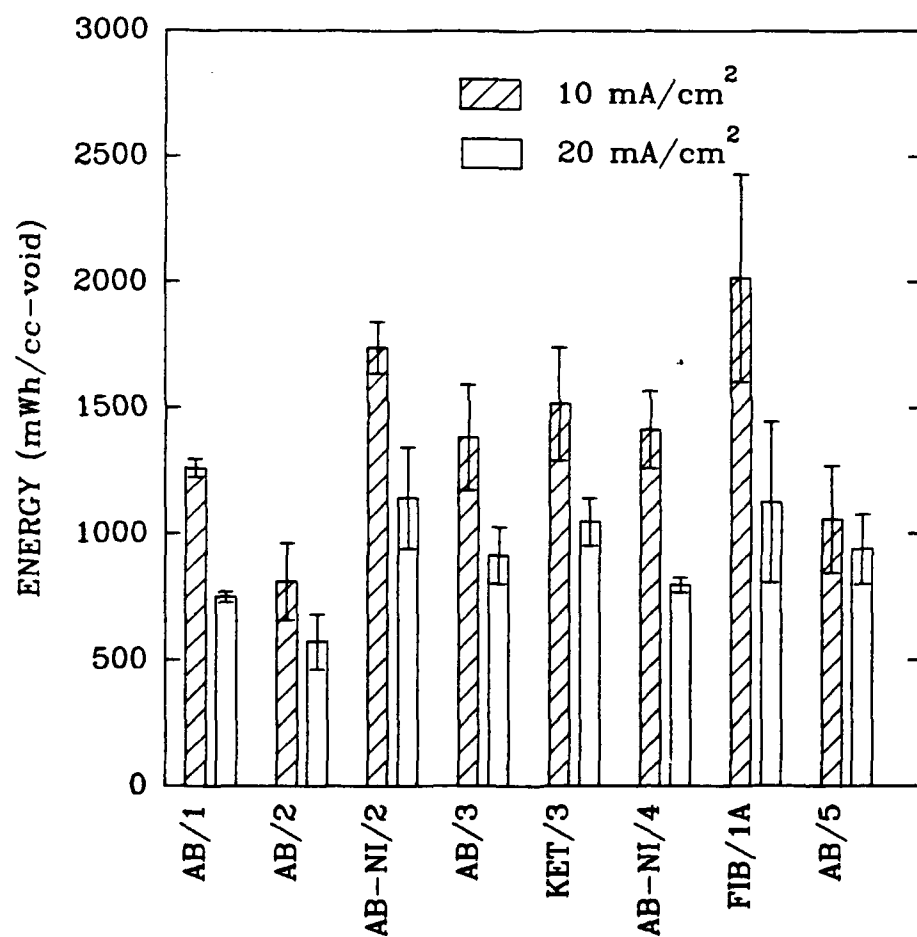


Fig. 8a

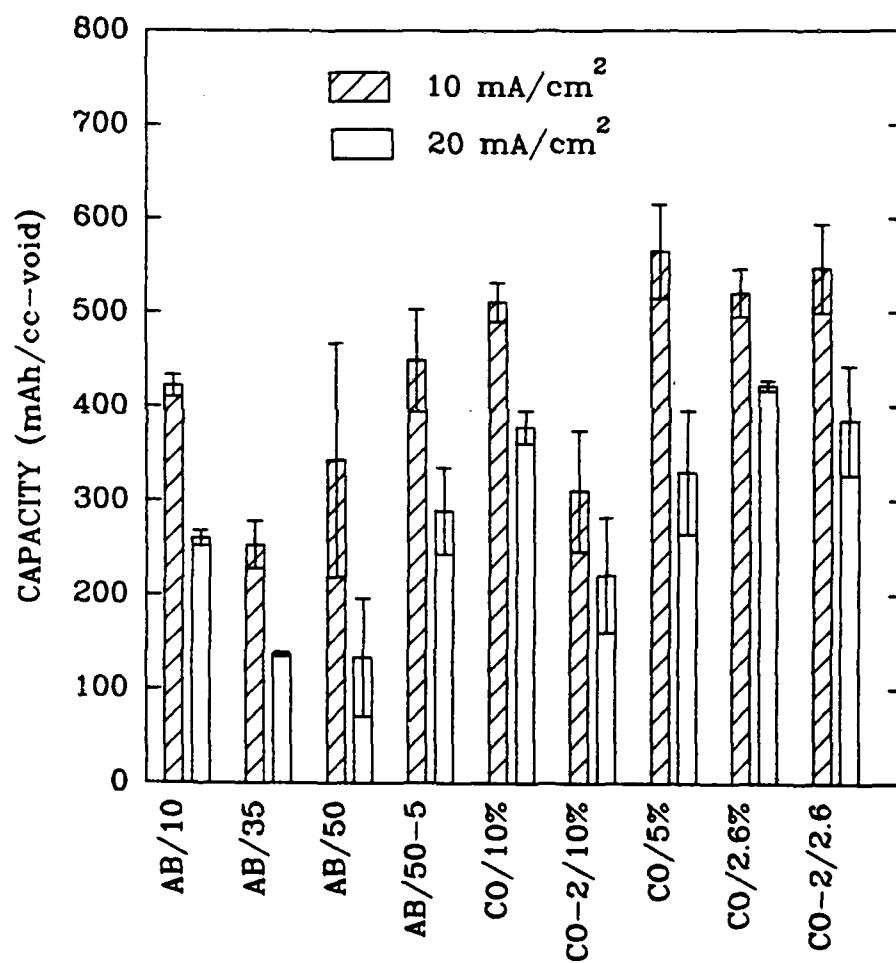


Fig. 8b.

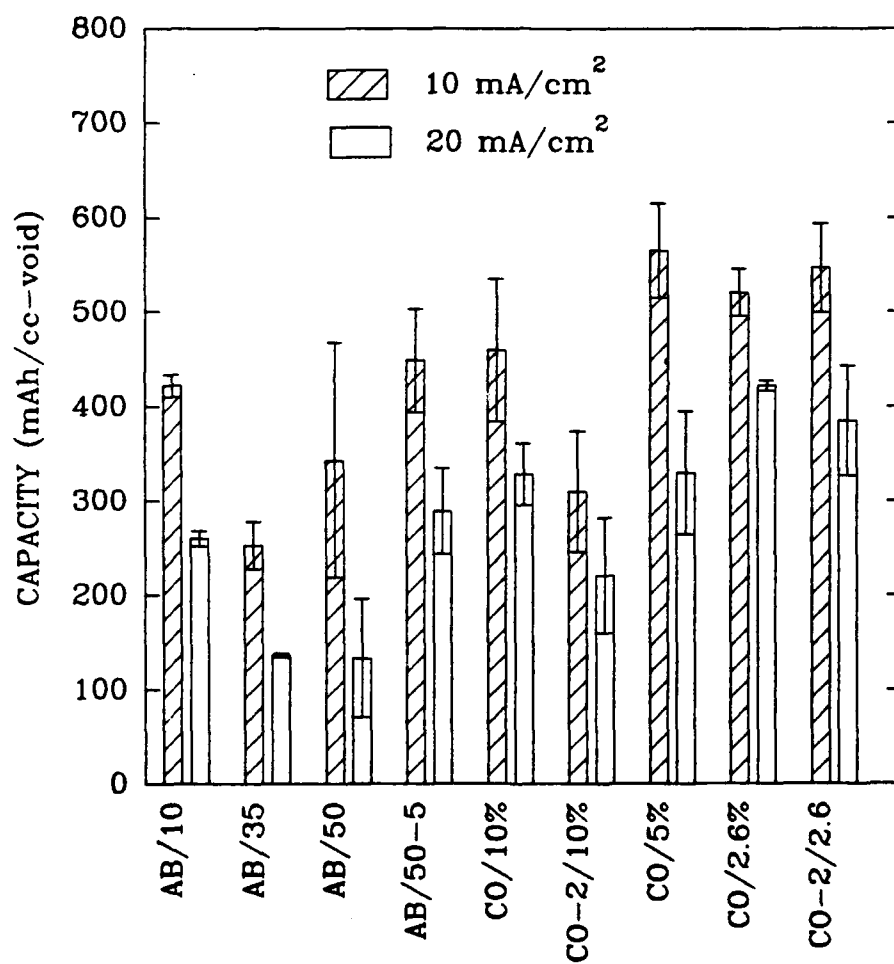


Fig 9

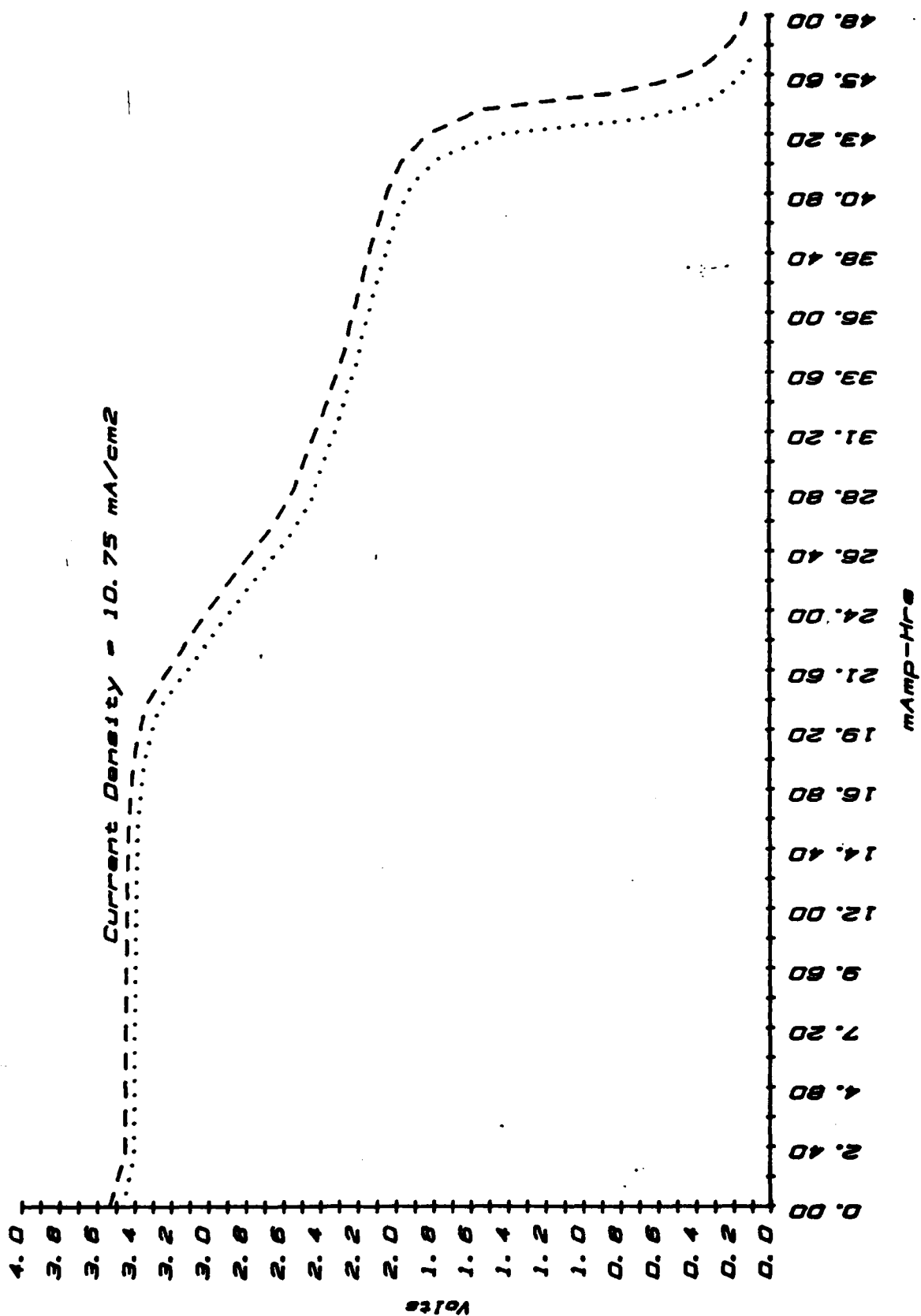


Fig 10

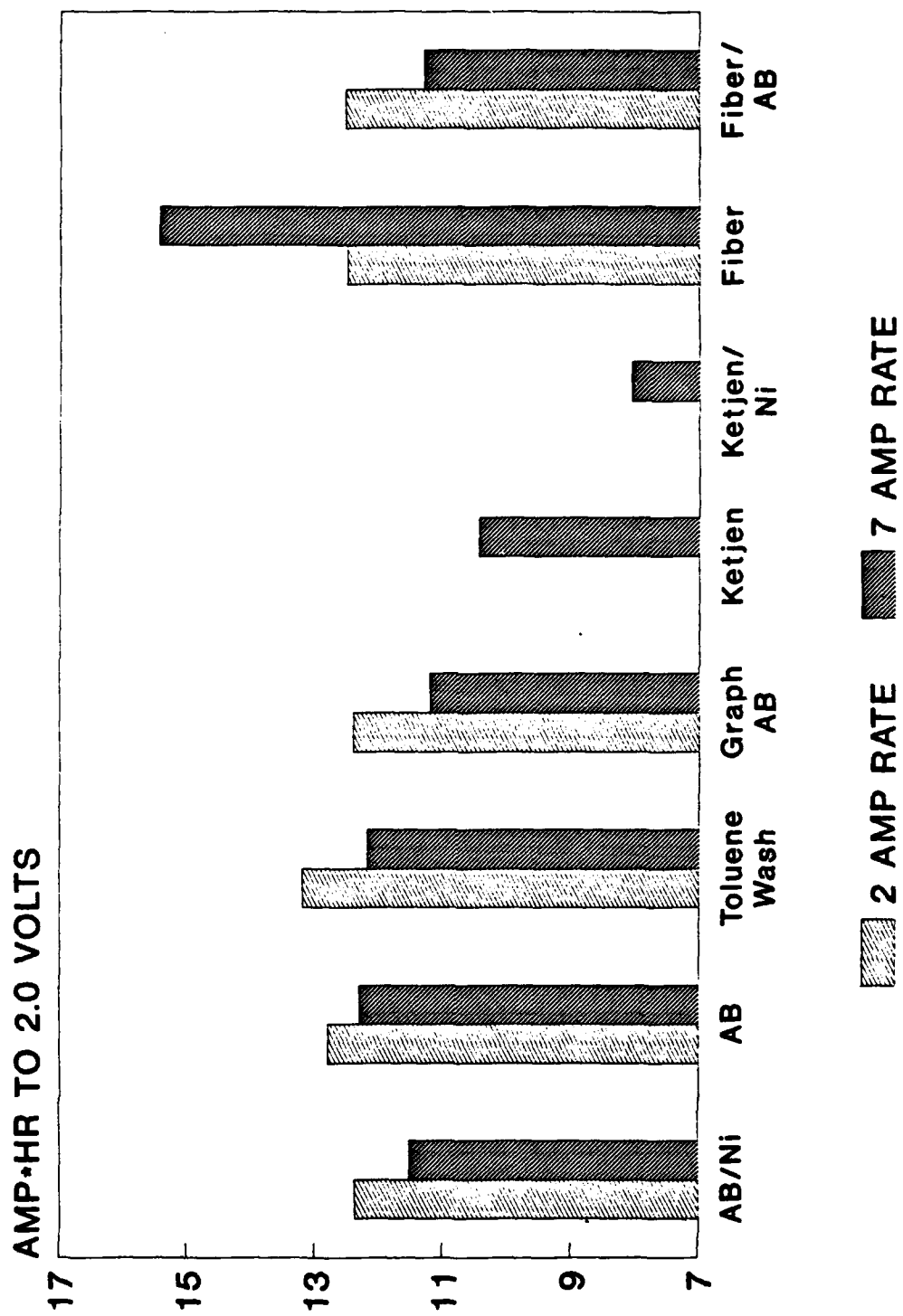


Fig. 11

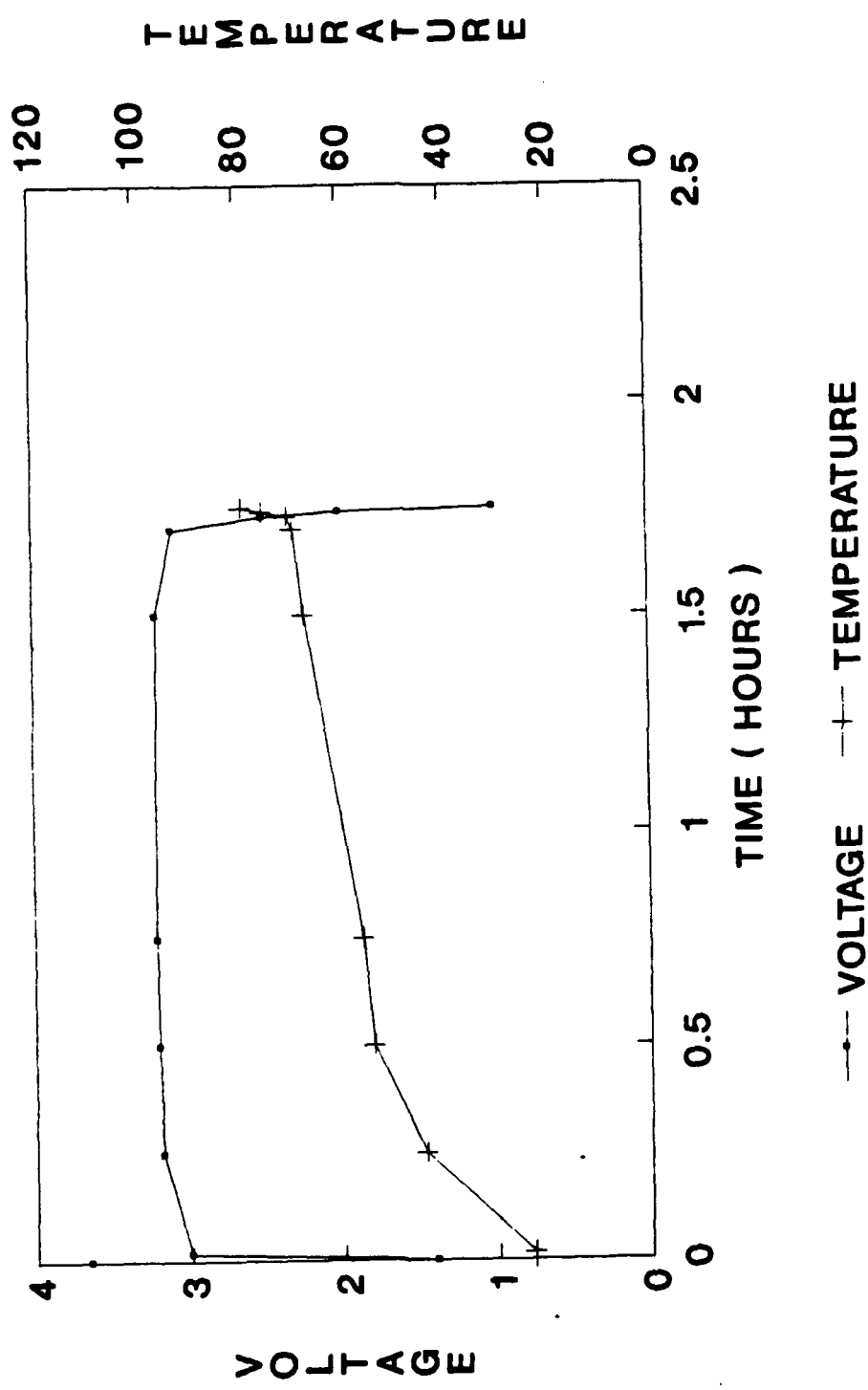


Fig. 12

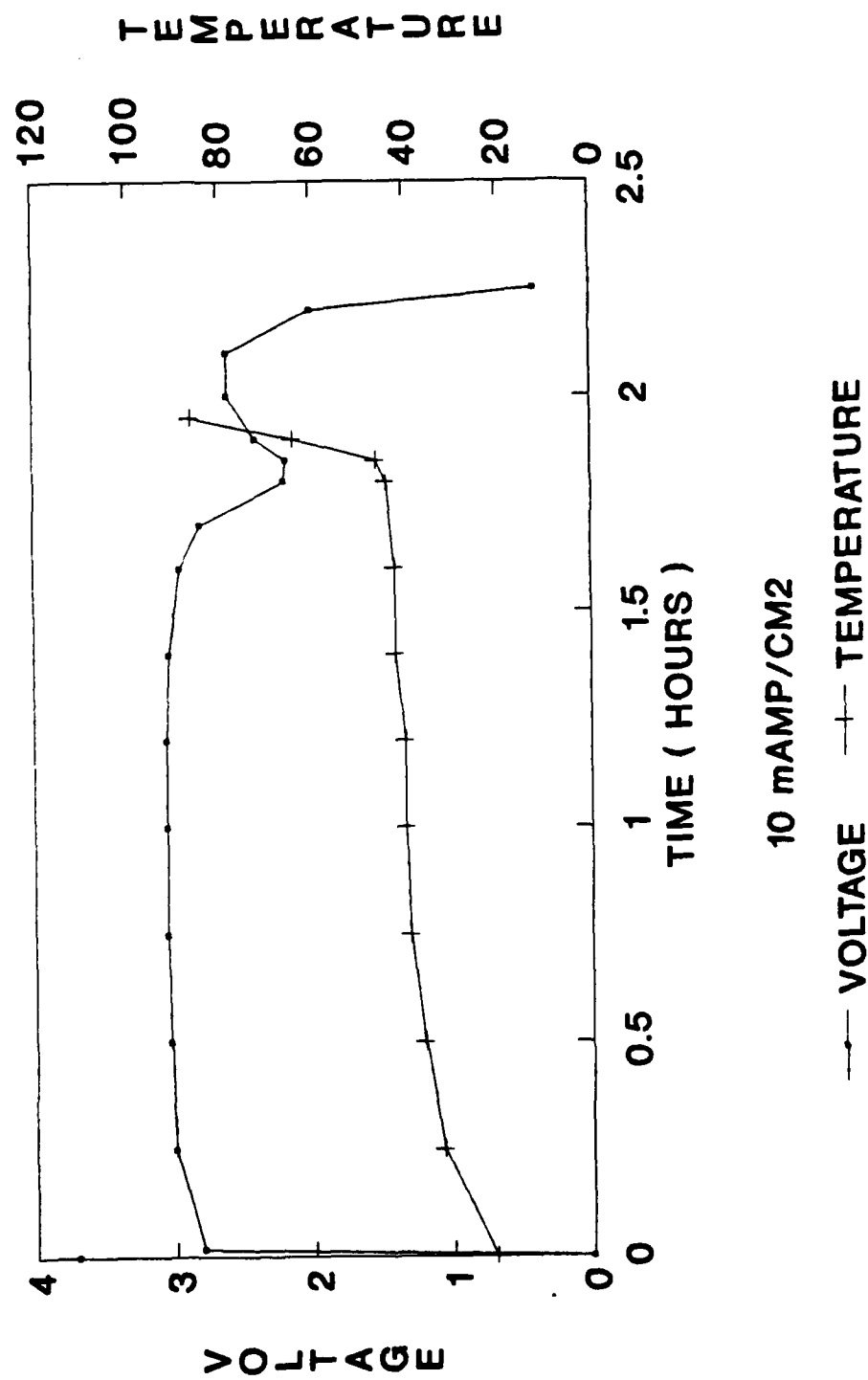




Fig 13

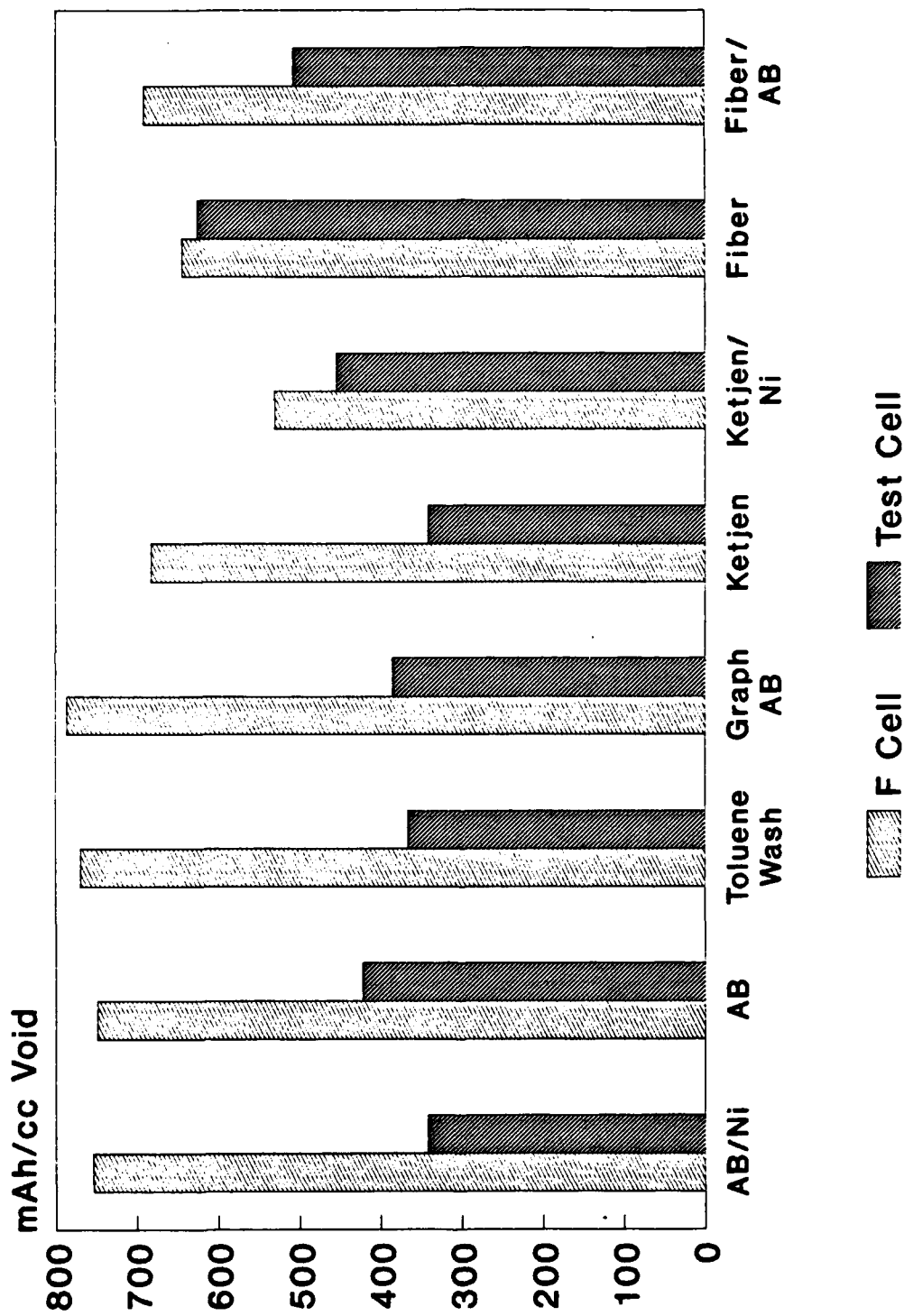
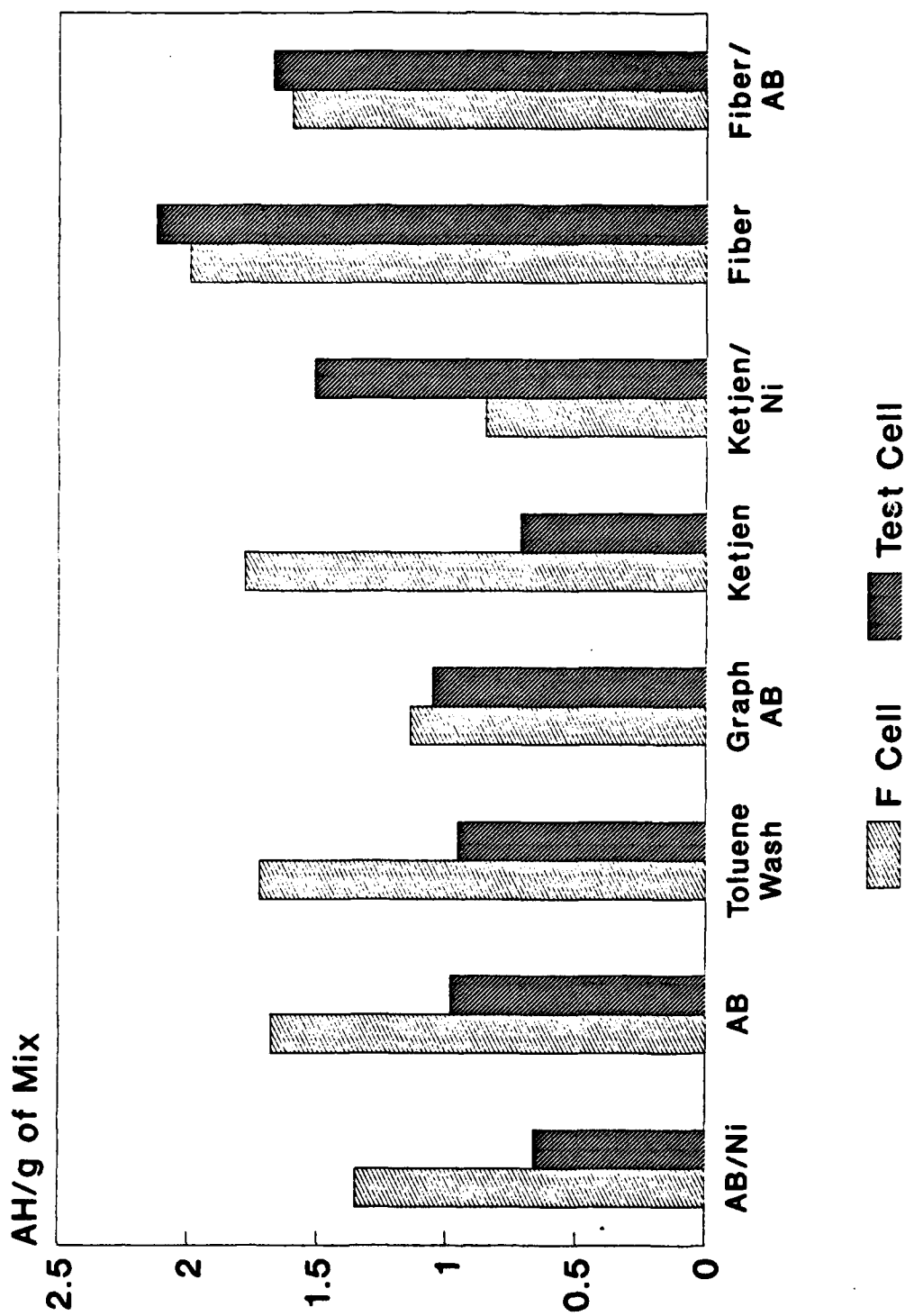


Fig. 14



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